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Indian Standard

METHODS OF TESTS FOR OXIDATIVE AGEING OF ELECTRICAL INSULATING PETROLEUM OILS BY OPEN BEAKER METHOD

(First Reprint JULY 1999)

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**AMENDMENT NO. 1 DECEMBER 2010
TO
IS 12177 : 1987 METHODS OF TESTS FOR
OXIDATIVE AGEING OF ELECTRICAL
INSULATING PETROLEUM OILS BY
OPEN BEAKER METHOD**

(Page 4, clause **0.4**) — Delete.

(Page 4, clause **0.5**) — Substitute '**0.4**' for '**0.5**'.

(Page 4, clause **0.5.1**) — Substitute '**0.4.1**' for '**0.5.1**'.

(Page 4, footnotes) — Delete the following footnote:

'§Specification for new insulating oils (*third revision*).'

(Page 5, clause **0.6**) — Substitute '**0.5**' for '**0.6**'.

(Page 7, clause **4.2**, line 2) — Substitute '2003' for '1973'.

(Page 7, clause **4.10**, line 2) — Delete 'These tests should be carried out on unaged samples as well since this will facilitate assessment of the effect of ageing.'

(Page 7, footnotes) — Add '(first revision)' after '†Method of sampling for liquid dielectrics'.

(Page 8, clause **6**) — Delete the words '**AND ACCURACY**'.

(Page 8, clause **7.3**, line 1) — Delete the words 'and unaged'.

(Page 9, Appendix A, clause **A-7**, line 1) — Substitute 'sludge to two significant figures' for 'percentages below 1 to three digits and percentages of 1 or more to two digits'.

(Page 9, Appendix B, Sl No. 2) — Substitute ' $60 \times 60 \times 60 \text{ cm}^3$ ' for ' $60 \times 60 \times 65 \text{ cm}^3$ '.

Amend No. 1 to IS 12177 : 1987

(Page 9, Appendix B, Sl No. 4, line 3) — Delete the following:

'Baking at elevated temperature ensures a hard coat which is much more scratch and wear resistant than the ordinary air drying paints.'

(ET 03)

Indian Standard

METHODS OF TESTS FOR OXIDATIVE AGEING OF ELECTRICAL INSULATING PETROLEUM OILS BY OPEN BEAKER METHOD

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(Continued on page 2)

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Indian Standard

METHODS OF TESTS FOR OXIDATIVE AGEING OF ELECTRICAL INSULATING PETROLEUM OILS BY OPEN BEAKER METHOD

0. F O R E W O R D

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 8 September 1987, after the draft finalized by the Liquids and Gaseous Dielectrics Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 It is generally recognized that all the methods have been designed to approximate the range of conditions under which the liquid filling is expected to operate in power transformers, primarily because oxygen exposure, although with significant variations due to the particular oil preservation system, is a common feature of transformer applications.

Insulating liquids, however, are also used as components of the insulation system in other items of equipment — typically cables, capacitors and bushings — where hermetic sealing after a high degree of degassing substantially eliminates the oxidation reaction in normal service. Oxygen exposure may only occur during manufacture or from smaller concentrations of residual air. Also, the increase in dielectric losses accompanying the oil oxidation may be most important in these applications.

A different but somewhat related aspect is the problem of system compatibility. Whilst it is known that oil ageing in the absence of oxygen is an extremely slow process, it has been sometimes reported that elevated temperature in association with system metals may induce instability and degradation of dielectric properties.

0.2.1 It is expected to test the oil for electrical characteristics besides those of acidity and sludge after ageing. The test for oxidation stability checks the quality of oil. This standard gives the method of oxidation stability of insulating petroleum oils by open beaker method with metal catalyst and without a metal catalyst.

0.2.2 Open-beaker oxidative ageing methods have been used for many years in laboratories of oil companies, electrical equipment manufacturers and electric utility companies to check the stability of electrical insulating oils under oxidative conditions.

0.2.3 Specification limits for oils subjected to open beaker oxidative ageing by this method are established by agreement between the purchaser and the supplier of applicable type oils. The properties of oil before and after ageing to be determined are:

- a) specific resistance (resistivity),
- b) power factor,
- c) acidity, and
- d) sludge.

These properties are determined by appropriate test methods as given in IS : 1448 (Part 2)-1967* for total acidity, IS : 6103-1971† for test for specific resistance (resistivity) and IS : 6262-1971‡ for test for dielectric dissipation factor. The procedure for determining total sludge value is given in Appendix A.

0.3 This standard applies to the evaluation and quality control of unused oils either inhibited or uninhibited, used as impregnating or pressure media as long as less than 10 percent of the oils evaporates during the ageing procedure.

0.4 Appendix C of IS : 335-1983§ shall be withdrawn after publication of this standard.

0.5 In the preparation of this standard, considerable assistance has been derived from the following:

ASTM D 1934-1968 Standard method for oxidative ageing of electrical insulating petroleum oils by open-beaker methods. American Society for Testing and Materials.

ASTM E 145-68 (1981) Specification for gravity-convection and forced-ventilation ovens. American Society for Testing and Materials.

BS : 2648-1955 Performance requirements for electrically-heated laboratory drying ovens. British Standards Institution.

0.5.1 Assistance has also been derived from IEC Doc : 10A (Secretariat) 95 Secretariat enquiry concerning the oxidation stability of insulating liquids for applications in air-tight equipment such as cables and capacitors, issued by the International Electrotechnical Commission.

*Methods of test for petroleum and its products: Part 2 Acidity (*first revision*).

†Methods of test for specific resistance (resistivity) of electrical insulating liquids.

‡Method of test for power factor and dielectric constant of electrical insulating liquids.

§Specification for new insulating oils (*third revision*).

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This test method covers procedure for subjecting electrical insulating oils to oxidative ageing by:

Method A — with metal catalyst

Method B — without metal catalyst.

1.2 This method is applicable to electrical insulating oils of petroleum origin as long as less than 10 percent of the oil evaporates during the ageing procedures. At present, it applies and is generally useful primarily in the evaluation and quality control of unused oils, either inhibited or uninhibited.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply:

2.1 **Total Sludge Value** — The percentage by weight of insoluble matter formed when the oil is heated and oxidized under specified conditions and subsequently diluted with n-heptane.

2.2 **Total Acidity** — It is a measure of free organic and inorganic acids present together and is expressed as milligrams of potassium hydroxide required to neutralize the total free acids in one gram of oil.

2.3 **Specific Resistance (Resistivity)** — It is the ratio of the dc potential gradient in volts per centimetre paralleling the current flow within the specimen to the current density in amperes per square centimetre at a given instant of time and under prescribed conditions. This is numerically equal to the resistance between opposite faces of a centimetre cube of the liquid. It is expressed in ohm-centimetre.

2.4 **Dielectric Dissipation Factor (Tan Delta)** — It is the tangent of the angle (delta) by which the phase difference between applied voltage and

*Rules for rounding off numerical values (revised).

resulting current deviates from $\pi/2$ radian, when the dielectric of the capacitor consists exclusively of the insulating oil.

2.5 Repeatability — A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

2.6 Reproducibility — A quantitative measure of variability associated with operators working in different laboratories, each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run only in one case in twenty in the normal and correct operation of the test method.

2.7 Oxidative Ageing — Is considered to be the exposure of oil to oxygen under certain specified conditions.

2.8 Copper Catalyst — Is a metal that either increases the rate of oxidation of the oil or reacts with the oxidation products to increase oil dielectric loss.

3. APPARATUS

3.1 Oven

3.1.1 Oven should be electrically heated thermostatically controlled, capable of maintaining a constant temperature (*see* Appendix B)

3.1.2 During the ageing period, air shall be circulated in the chamber by a low-velocity fan.

3.1.3 The volume and condition of the circulated air is not considered to be critical. The oven (as given in Appendix B) should be equipped with a slowly rotating shelf. However, it is recommended that the oven provides several volume changes per hour and that vapours and fumes be removed.

3.2 Beaker — Made of borosilicate glass, low-form of 400 ml capacity. The approximate dimensions of a suitable beaker are 100 mm deep and 70 mm inside diameter.

3.3 Copper Catalyst — Copper catalyst with 15 cm² of surface available for exposure to the oil for use in Method A. The dimensions of the copper

catalyst shall be 475 ± 2 mm length of electrolytic copper wire of diameter 1.00 to 1.02 mm is rolled into a spiral of approximately 30 mm external diameter and 50 mm in height.

4. PROCEDURE (METHOD A)

4.1 Clean the beaker thoroughly to remove residual oil by rinsing in mineral spirit. Rinse thoroughly with an acid cleaning solution consisting of 3 parts nitric acid (HNO_3) to 1 part sulphuric acid (H_2SO_4). Remove traces of cleaning solution by carefully rinsing with distilled water (*see* IS : 1070-1977*) followed by rinsing with acetone and air drying.

4.2 Obtain the oil samples by appropriate procedure given in IS : 6855-1973†.

4.3 Adjust the oven temperature to $115 \pm 1^\circ\text{C}$.

4.4 Pour without preheating 300 ml of the sample to be tested into a clean dry 400-ml beaker. Oil depth in the beaker will be approximately 75 mm.

4.5 Insert the copper catalyst described in 3.3 in the oil samples in the beaker.

4.6 Place the beaker containing the sample in the preheated oven. To minimize temperature fluctuation, it is desirable to place all samples in the oven at the same time.

4.7 Start the oven shelf into rotation.

4.8 Remove the sample from the oven at the end of the 96-h ageing period.

4.9 Transfer the aged sample from the beaker to a suitable glass container and tightly closed to prevent the sample from being exposed to atmospheric air.

4.10 Use the aged oil samples for determination of total sludge, neutralization value, dielectric dissipation factor and specific resistance. These tests should be carried out on unaged samples as well since this will facilitate assessment of the effect of ageing.

*Specification for water for general laboratory use (*second revision*).

†Method of sampling for liquid dielectrics.

5. PROCEDURE (METHOD B)

5.1 Procedure given in 4.1 to 4.10 except 4.5 in Method 'A' shall be followed for oxidative ageing without metal catalyst.

6. PRECISION AND ACCURACY

6.1 Repeatability and reproducibility have not been established as experiments are still being conducted in different laboratories.

7. REPORT

7.0 The report shall include the following.

7.1 Whether or not a metal catalyst was used (Method A or B).

7.2 Sample identification.

7.3 Values of the properties measured on the aged and unaged samples.

A P P E N D I X A
(*Clause 0.2.3*)

A-1. The original sample shall be used for the determination of total acidity, specific resistance and dielectric dissipation factor.

A-1.1 Another sample shall be used for the determination of total sludge.

A-2. Dilute the other sample to 1 000 ml with n-heptane.

A-2.1 Allow it to stand overnight, or approximately 16 h at ambient temperature.

A-3. Filter the oil-heptane solution through a previously dried, cooled and weighed G 4 glass filter making certain that all solid material is transferred to the filter by thoroughly cleaning and washing the container with n-heptane.

A-4. Wash the sludge in the crucible thoroughly with n-heptane to remove all traces of oil.

A-5. Dry the sludge in the filter in an oven at 110°C to constant weight. Cool it in a desiccator and weigh to the nearest 0.000 1 g.

A-6. Sludge, percentage = $\frac{100 \times (a - b)}{300 \times \text{density of oil}}$

where

a = weight of sludge and crucible in grams, and

b = weight of crucible in grams.

A-7. Report percentages below 1 to three digits and percentages of 1 or more to two digits.

APPENDIX B

(*Clauses 3.1.1 and 3.1.3*)

SPECIFICATION FOR LABORATORY OVEN WITH ROTATING SHELVES FOR OXIDATIVE AGEING TESTS ON INSULATING OILS

- | | |
|---------------------------|---|
| 1. Scope | This specification covers requirements of electrically heated forced oven for laboratory evaluation of insulating oils. This specification is generally based on ASTM-E-145-IIA IS : 6365-1971 Specification for laboratory electric ovens and BS 2648 : 1955 Performance requirements for electrically heated laboratory drying ovens. |
| 2. Dimensions of the oven | $60 \times 60 \times 65 \text{ cm}^3$ or $45 \times 45 \times 45 \text{ cm}^3$ |
| 3. Inner chamber | Made of steel and duly reinforced for mechanical strength. |
| 4. Outer chamber | Made of mild steel or stainless steel and duly reinforced for mechanical strength. Baking at elevated temperature ensures a hard coat which is much more scratch and wear resistant than the ordinary air drying paints. |
| 5. Temperature range | Ambient temperature up to 300°C maximum. |
| 6. Temperature control | Suitable temperature controller to be provided. |

IS : 12177 - 1987

- | | |
|--|---|
| 7. Temperature read out | Suitable temperature indicating device. |
| 8. Insulation | 76 mm glass wool layer or equivalent. |
| 9. Temperature accuracy | Plus or minus 1°C, maximum in the range 100-200°C. |
| 10. Rate of ventilation | Adjustable over the entire required range. |
| 11. Air circulation | Suitable forced air circulation. |
| 12. Safety features | Thermal safety cut-out should be at slightly higher temperature than the set temperature. |
| 13. Input | Should be designed for supply voltage of 230 ± 10 V percent, single phase 50 Hz. |
| 14. Number of shelves | Two |
| 15. Rate of rotation or
speed of shelves,
<i>Min</i> | 1 ppm. |

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